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(54) Title: LYOCELL FIBRE TREATMENT

(57) Abstract

The absorbency of lyocell fibre can be increased by scouring in hot aqueous alkali, preferably in loose state, for example in 1-6 % sodium hydroxide at 90-125 °C for 1-10 hours.

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LYOCELL FIBRE TREATMENT

Field of the invention

This invention relates to methods of increasing the absorbency of lyocell fibre.

It is known that cellulose fibre can be made by extrusion of a solution of cellulose in a suitable solvent into a coagulating bath. This process of extrusion and coagulation is referred to as "solvent-spinning", and the cellulose fibre produced thereby is referred to as "solvent-spun" cellulose fibre or under the generic name lyocell fibre. One example of such a process is described in US-A-4,246,221, the contents of which are incorporated herein by way of reference. Cellulose is dissolved in a solvent such as an aqueous tertiary amine N-oxide, for example aqueous N-methylmorpholine N-oxide. The resulting solution is extruded through a suitable die to produce filaments, which are coagulated, washed in water to remove the solvent and dried. The filaments are commonly cut into short lengths at some stage after coagulation to form staple fibre. It is also known that cellulose fibre can be made by extrusion of a solution of a cellulose derivative into a coagulating and regenerating bath. One example of such a process is the viscose process, in which the cellulose derivative is cellulose xanthate. Both such types of process are examples of wet-spinning processes. Solvent-spinning has a number of advantages over other known processes for the manufacture of cellulose fibre such as the viscose process, for example reduced environmental emissions.

As used herein, the term "lyocell fibre" means a cellulose fibre obtained by an organic solvent-spinning process in which the organic solvent essentially comprises a mixture of organic chemicals and water and in which solvent-spinning involves dissolving cellulose and spinning without formation of a derivative of the cellulose. As used herein, the terms "solvent-spun cellulose fibre" and "lyocell fibre" are synonymous.

Cellulose is a naturally hydrophilic and absorbent material. There is nevertheless a desire for lyocell fibre having increased absorbency for use in absorbent products such as swabs and tampons.

Background art

Natural cellulose fibres such as cotton are commonly subjected to the process called scouring in order to dissolve natural impurities such as protein, pectin, ash and wax, which occur together with cellulose in such natural fibres. Scouring processes are described for example by A J Hall in Chapter 4 of The Standard Handbook of Textiles, Newnes Butterworth, 8th edition (1975). In particular, cotton which is to be used as an absorbent must be thoroughly scoured under severe conditions in order to remove hydrophobic waxy impurities whose presence adversely affects the absorbency of the fibre. It is well-known that man-made cellulose fibres, including lyocell fibres, do not contain such hydrophobic impurities and accordingly do not require such severe scouring as cotton, although they are commonly scoured at late stages in processing in order to remove spin-finishes and the like. It is further well-known that regenerated cellulose fibres such as viscose rayon fibres are more sensitive to alkali than cotton is and that they should generally be scoured under as mild conditions as possible to avoid loss of mechanical properties or even total destruction.

Disclosure of the invention

According to the invention there is provided a method of increasing the absorbency of lyocell fibre, characterised in that the fibre is scoured in a hot aqueous solution of alkali. The method of the invention is preferably performed on loose fibre.

The method of the invention is generally carried out at a temperature of at least 90°C, preferably in the range from

90 to 125°C. It is preferably carried out at the boil in an open kier or at higher temperatures in a pressure kier. The treatment time may generally be in the range from 1 to 10 hours. It will be understood that lower temperatures and shorter treatment times generally necessitate the use of more strongly alkaline scouring liquors.

The scouring liquor used in the method of the invention may be of similar composition to that of liquors known in the art for the thorough scouring of cotton. Such liquors contain sodium hydroxide as alkali, generally at a concentration of from 1 to 6, often from 2 to 5, percent by weight. Such liquors may additionally contain other alkalis such as sodium carbonate, especially if the scouring process is carried out in an open vessel, because inclusion of sodium carbonate is known to reduce the risk of yellowing of the cellulose under such conditions. Nevertheless, useful results can be obtained from the method of the invention when the scouring liquor contains from 0.5 to 20 percent by weight sodium hydroxide. The scouring liquor may also include one or more assistants such as those conventionally used in the scouring of cotton, for example wetting agents such as anionic and nonionic surfactants.

The reasons for the increased absorbency imparted by the method of the invention are not understood. It is noteworthy that the method of the invention has only a minor effect on the water imbibition of the fibre. It is also noteworthy, and surprising, that the method of the invention may reduce the total free absorbency (TFA) of the fibre and/or its absorbency rate (as assessed by sink time) but that nonetheless absorbent articles (such as tampons) made from the fibre exhibit increased absorbency. It is remarkable that the method of the invention increases the absorbency of lyocell fibres in that the untreated fibres contain very low proportions of non-cellulosic or hydrophilic impurities. It is further remarkable that lyocell fibres can be treated by the method of the invention without severe degradation of their physical

properties.

Lyocell fibre scoured by the method of the invention may subsequently be bleached using similar conditions to those known for cotton, for example using aqueous sodium hypochlorite. Bleaching may for example be performed using a solution containing from 1 to 10 g/l active chlorine and having a pH in the range from 9 to 11 at room temperature for from 1 to 10 hours. It may be found that bleaching initially further serves to increase the absorbency of the fibre but that more extended bleaching tends to reduce its absorbency.

The scouring and optional bleaching treatments may each be performed once or several times, for example two to four times. All such scouring treatments may be performed before all such bleaching treatments, or alternatively the scouring and bleaching treatments may be performed in alternation.

After the scouring and optional bleaching treatments, the lyocell fibre may be washed and dried in conventional manner. The washing process generally includes a wash with dilute aqueous acid, for example hydrochloric acid.

Lyocell fibre may be treated according to the method of the invention in the form of tow, filaments, loose staple fibre, yarns, fabrics or other articles, alone or in blend with other materials such as cotton. The decitex of the lyocell fibre is commonly in the range from 0.5 to 10.

Lyocell fibre treated according to the method of the invention may exhibit increased absorbency in comparison with untreated fibre and is suited for the manufacture of absorbent articles such as swabs and tampons having increased absorbency in comparison with articles made from untreated fibre. Fibre treated according to the method of the invention may be used in the manufacture of absorbent articles on its own or in blend with other fibres. The combination, in fibre treated according to the method of the invention, of a high degree of

absorbency with high or low rates of absorption permits the ready manufacture of absorbent articles of a controlled degree of absorbency and rate of absorption, either by suitable choice of treatment conditions or by blending.

Test Method 1 - Modified Syngina Test

The absorbency of cellulose fibres may be assessed by the following procedure, called the Modified Syngina Test. A well-blended sample of fibre weighing at least 30 g is opened or carded by hand and formed into a web using a Shirley miniature card. The carded web is stored in a conditioned atmosphere (20±2/C, 65±2% relative humidity RH) for 24 hours. The web is folded lengthways into three layers and cut to form a 100 mm x 45 mm pad weighing 2.72±0.05 g, in which the fibres run parallel to the long dimension of the rectangle. The pad is placed into a cross-die assembly and pressed at 6.9 MPa (1000 psig) for 60 seconds to form a longitudinally-expanding tampon of nominal length 20 mm and nominal diameter 15 mm having an average density of about 0.35 g/cm^3. The tampon is then stored in a conditioned atmosphere for 2 hours in conventional manner and its length measured. Tampons which have expanded to a length of more than 50 mm during this storage are rejected, and if necessary pressing conditions are adjusted to provide tampons with greater stability to expansion. Tampon absorbency was assessed using the test defined in GB-B-2,094,637 except that 180 mm hydrostatic head water pressure was employed, the Syngina chamber was tilted at 30/ to the vertical, and the 1% saline solution was injected into the head of the tampon, using a cannula, at a rate of 50 ml/hour. Three tampons are tested and the results, reported as grams of saline solution absorbed per gram of fibre (g/g), are averaged. Tampons made from a standard control sample of viscose rayon fibre are tested in each series of experiments to ensure reproducibility.

Test Method 2 - Sink Time and Total Free Absorbency

Sink Time and Total Free Absorbency (TFA) were measured by the methods described in British Pharmacopoeia 1988 (HMSO, 1988), Appendix XX L, page A226 under the titles "Absorbency - Sinking Time" and "Absorbency - Water Holding Capacity" respectively.

Test Method 3 - Water Imbibition

This was assessed by a method based on ASTM D2402-78. A weighed sample of fibre is thoroughly wetted with water and centrifuged at 2700-2800 g for 5 minutes. The centrifuged fibre is weighed, and water imbibition (W.I.) is calculated as the percentage by which the weight of the centrifuged sample exceeds that of the dry sample before testing.

The invention is illustrated by the following Examples, in which parts and proportions are by weight unless otherwise specified.

Example 1

Bright lyocell staple fibre (1.7 dtex 38 mm) was scoured and bleached in a variety of sequences. Scouring was performed using aqueous sodium hydroxide in an open vessel at the boil. Bleaching was effected using aqueous sodium hypochlorite containing 3.5 or 4.4 g/l active chlorine at room temperature.

Longitudinally-expanding tampons were made from 1.7 dtex 38 mm bright lyocell fibre (available from Courtaulds Fibres (Holdings) Limited), both scoured and unscoured, and from a control sample of 3.3 dtex 38 mm bright trilobal viscose fibre (available from Courtaulds Fibres Limited under the Trade Mark GALAXY and as described in EP-A-0,301,874). This trilobal viscose fibre is used in the commercial manufacture of tampons. The results given in Table 1 were obtained in the Modified Syngina Test, for TFA, and for W.I.

Table 1

Ref	Treatment conditions	Length after storage mm	Syngina absorbency g/g	TFA ml/g	W.I.
LC	Lyocell control	15	3.4	20	54-55
VC	Trilobal viscose control	16	5.0-5.4	24	90
1.	Bleach (3.5 g/l)	19	4.9	26	47
2.	2 x bleach (3.5 g/l)	20	5.5	26	48
3.	3 x bleach (3.5 g/l)	19	4.6	27	45
4.	Bleach (4.4 g/l)	20	5.1	26	48
5.	2 x bleach (4.4 g/l)	22	4.9	26	52
6.	Scour	19	5.6	30	56
7.	Scour - bleach (4.4 g/l)	19	5.3	31	52
8.	2 x scour	24	5.8	27	52
9.	2 x scour - bleach (4.4 g/l)	20	5.4	30	50
10.	2xscour- 2xbleach (4.4 g/l)	21	5.5	28	48
11.	Scour - bleach (4.4 g/l) - scour	22	5.2	29	52
12.	2 x (scour - bleach (4.4 g/l))	20	5.2	31	51

It can be seen that the scoured lyocell fibre exhibited an improved Syngina absorbency in all cases, in some cases surpassing that of the trilobal viscose control. Bleaching in the absence of scouring had a lesser and more variable effect.

Samples LC and 6 were also made into radially-expanding tampons of a rolled construction 2.8 g in weight. The wet diameter and absorbency results recorded in Table 2 were obtained:

Table 2

Ref	Wet diameter mm	Absorbency g/g
LC	21	4.1
6	25	4.8

Example 2

Lyocell staple fibre (1.7 dtex, 38 mm, crimped, soft finish) was scoured in a package dyeing machine under various conditions. In this machine, the scouring liquor is caused to flow through a closed vessel containing the fibre. At the end of the treatment, the scouring liquor is drained from the system and the fibre washed with hot water until the effluent is free of alkali. Further details and experimental results are shown in Table 3:

Table 3

NaOH conen %	Time hr	Temp °C	Stability Length mm	Syngina Absorbency g/g Longitudinally-expanding	Syngina Absorbency g/g Radially-expanding	TFA ml/g	Sink Time	W.I.	Tenacity (cN/tex)	Extension (%)	Decitex
Control	-	-	14	3.65	3.57	20.4	3.7	56	41.5	14.5	1.8
0.5	1	100	15	4.68	4.29	22.7	6.2	53	40.7	12.1	1.8
0.5	5	115	15	4.91	4.45	11.9	21.5	52	39.1	11.1	1.8
2.0	1	115	15	4.89	4.53	7.1	21.1	54	41.1	11.8	1.8
2.0	5	100	15	4.86	4.64	19.4	20.7	55	37.9	10.4	1.7
10	1	100	18	4.19	4.43	19.8	3.6	62	34.9	13.8	1.7
15	1	100	20	4.34	4.04	19.4	3.2	59	23.2	11.2	1.8
20	1	100	17	4.36	4.04	21.2	3.5	58	25.2	9.6	1.7

All the scoured samples of lyocell fibre exhibited increased absorbency when made into tampons, in spite of the observations that total free absorbency was in some cases markedly reduced and/or sink time was markedly increased and that there was little significant change in W.I.

CLAIMS

1. A method of increasing the absorbency of lyocell fibre, characterised in that the fibre is scoured in a hot aqueous solution of alkali.

2. A method according to claim 1, further characterised in that the fibre is scoured in the form of loose fibre.

3. A method according to one of claim 1 or claim 2, further characterised in that the temperature of the solution is at least 90°C.

4. A method according to claim 3, further characterised in that the temperature of the solution is in the range from 90 to 125°C.

5. A method according to any one of the preceding claims, further characterised in that scouring is carried out for a time in the range from 1 to 10 hours.

6. A method according to any one of the preceding claims, further characterised in that the alkali is sodium hydroxide.

7. A method according to claim 6, further characterised in that the concentration of sodium hydroxide in the solution is in the range from 1 to 6 percent by weight.

8. A method according to any one of the preceding claims, further characterised in that the fibre is scoured from two to four times.

9. A method according to any one of the preceding claims, further characterised in that the fibre is subsequently bleached with aqueous sodium hypochlorite.

10. A method according to claim 9, further characterised in that the fibre is bleached from two to four times.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 D06M11/38 D06M11/30

According to International Patent Classification(IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 D06M D01F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category '	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A,P	WO 97 23668 A (COURTAULDS FIBRES HOLDINGS LTD ;BERTRAM DAVID (GB); GRAVESON IAN ( ) 3 July 1997 see page 2, line 6 - page 3, line 28 see page 4, line 30 - page 5, line 3 see page 5, line 30 - line 34 see examples ---	1-8
A	WO 95 24524 A (COURTAULDS FIBRES HOLDINGS LTD ;TAYLOR JAMES MARTIN (GB)) 14 September 1995 see page 3, line 7 - page 5, line 19 see examples 1,2 --- -/--	1-8

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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A	CHEMICAL ABSTRACTS, vol. 115, no. 6, 12 August 1991 Columbus, Ohio, US; abstract no. 51807, ANDRZEJEWSKI, SLAWOMIR ET AL: "Continuous or semicontinuous oxidative desizing and bleaching of cellulosic and cellulosic-synthetic blend fabrics using aqueous sodium hypochlorite solutions" XP002052123 see abstract & PL 152 402 A (CENTRALNY OSRODEK BADAWCZO-ROZWOJOWY PRZEMYSLU BAWELNIANEGO, POL.) ---	1-10
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